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DOI: https://doi.org/10.1080/01694243.2019.1602903

Originally published at:
Mangione, Elisabetta; Özcan, Mutlu (2019). Adhesion of resin cements to contaminated zirconia resin cements on zirconia: effect saliva-contamination and surface conditioning. Journal of Adhesion Science and Technology, 33(14):1572-1583.
DOI: https://doi.org/10.1080/01694243.2019.1602903

Adhesion of resin cements to contaminated zirconia resin cements on zirconia: effect saliva contamination and surface conditioning

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Short title: *Adhesion of resin cements to contaminated zirconia*

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Abstract: This study evaluated the adhesion of resin cements to zirconia after saliva contamination using resin cements with different chemistries. Zirconia discs (N=240, n=10 per group) were randomly divided into 3 groups: a) C: No contamination (Control), b) S: Contamination with saliva, c) S+AA: Contamination with saliva followed by air-abrasion (CoJet). While half of the specimens were not conditioned, the other half were conditioned with 37.5% H_3PO_4 for 60 s. After rinsing, all specimen surfaces were silanized (Monobond Plus). Resin cements based on either methacrylate (Variolink II-VL) or MDP monomer (Panavia 21-PN) were polymerized on the substrates. The specimens were randomly divided into two further groups to be tested either after a) 24 h dry storage at 37°C or b) thermocycling (x5000, 5-55°C). Microshear bond (MSB) tests were conducted in a Universal Testing Machine and failure types were analyzed. Data were analyzed using Univariate analysis and Tukey`s tests (alpha=0.05). While saliva contamination, 37.5% H_3PO_4 application ($p<0.001$) and aging ($p<0.05$) significantly affected the bond results, cement type did not show significant difference after aging ($p>0.05$). Adhesive strength of PN (1.2-4.4 MPa) on saliva contaminated and etched zirconia was more stable than that of VL (0-2.8 MPa). After aging, bond strength results decreased the most with VL (3 to 100%) compared to PN (32 to 71%) but the decrease was less in the air-abraded groups after aging (VL: 3%; PN: 32%). Exclusively adhesive failures were experienced in all groups.

Keywords: Adhesion; Bond Strength Test; Resin cements; Phosphoric acid; Saliva contamination; Surface conditioning; Surface contamination; Zirconium dioxide

Introduction

Ytrium stabilized polycrystalline zirconia (hereafter: zirconia) offers a wide variety of clinical applications, such as full coverage single crowns, fixed-dental-prosthesis (FDPs), resin-bonded FDPs, root posts or implant abutments in reconstructive dentistry. Zirconia has the most favorable properties compared to other high-strength ceramics having flexural strength of 900 to 1200 MPa, fracture resistance of more than 2000 N and fracture toughness of 9-10 MPa/mm^2 that is almost twice the value for alumina-based materials and almost three times higher then that of lithium disilicate-based ceramics [1]. With the advances in adhesive promoters, during the last few decades, indication of resin-bonded minimal invasive restorations increased substantially in reconstructive dentistry. In that respect, not only the strength of the restorative material but also the adhesion of resin-based luting cements both to the dental tissues and the particular restorative material is of importance for the long-term clinical success [2,3]. This aspect becomes even more important when retention of FDPs does not rely on macro-mechanical principles as in the case of resin-bonded surface-retained or cantilever FDPs [2,3].

Although etching with hydrofluoric acid and subsequent silanization of the cementation surface of glassy matrix ceramics is an efficient method to achieve durable adhesion of resin-based materials [4,5], neither etching with hydrofluoric nor applying silane coupling agents resulted in adequate adhesion to zirconia [4,6-10]. Since such ceramics do not contain a silicon dioxide (silica) phase, in order to enhance the adhesion of luting cements to oxide-based ceramics, a number of surface conditioning methods have been suggested [10]. While some of these methods facilitate resin-ceramic bonding micro-mechanically employing air-borne particle abrasion with alumina particles [8,10], others are based on physico-chemical activation of the ceramic surfaces using silica-coated alumina particles ranging in size from 30 to 250 μm followed by silanization [5,10,11] or chemical activation with functional monomer containing cements [10,11]. Chemical activation of zirconia is also possible using various silane coupling agents, primers and/or luting agents based on phosphate ester monomer 10-methacryloyloxydecyl dihydrogenphosphate (10-MDP), 4-methacryloxyethyl trimellitic anhydride (4-META), thiophosphoric acid methacrylate (MEPS) that react with oxides on zirconia [14-16]. However, all these cements or adhesion promoters still require air-abrasion in order to achieve a clean surface prior to their application according to the manufacturers. Such primers based on MDP also suffer from hydrolytic degradation [17]. Current state of art on adhesion to zirconia is that among all conditioning methods, particle deposition especially with silica-coated alumina particles delivers the most favourable adhesion [18,19].

Surface conditioning of zirconia surfaces could be performed either at chairside by the dentist or in the dental laboratory by the dental technician [20]. When performed by the dental technician, the subsequent try-in procedure leads to surface contamination with saliva and its proteins [21]. Saliva contains organic materials such as salivary proteins, enzymatic molecules, bacteria and food debris, and inorganic compounds such as mineral ions in water [22]. Adhesion of salivary proteins to dental materials and tooth surfaces, result in formation of acquired enamel pellicle that is free of bacteria at a thickness of 10-20 nm [23]. With the increase in the protein transmission from saliva, the thickness of this protein layer reaches to 100-1000 nm between 30-90 minutes [23]. The resulting persistent protein contamination from saliva in particular was shown to hinder adhesion of the resin cements [24,25].

Several cleaning methods have been proposed to eliminate the contamination from the ceramic surfaces, one of which is etching with H_3PO_4 [26-28]. Phosphoric acid removes the adsorbed proteins by coagulation or desorption from ceramic surface onto the cleaning particles. Subsequent water rinsing can then remove the coagulated or desorbed proteins in glassy matrix ceramics. Despite the fact that this method is easy to apply, H_3PO_4 could passivate and thereby inhibit the adhesion of MDP based cements to zirconia [29,30].

The objectives of this study therefore, were to evaluate the adhesion to zirconia after saliva contamination using resin cements with different chemistries and cleaning methods with and without aging. The null hypothesis tested was that bond strength of resin cements would not show significant difference depending on the cleaning method.

Materials and Methods

Specimen preparation

The brands, chemical compositions, manufacturers and batch numbers of the materials used in this study are listed in Table 1.

Zirconia specimens (N=240, n=10 per group) (Metoxid Dental, Thayngen, Switzerland) were prepared according to the manufacturer's recommendations (diameter: 10 mm; height: 2 mm). The specimens were wet ground finished using silicone carbide papers in sequence (# 400, 600, 800, 1200, 1500, 2000) for 30 s each. After sintering, the specimens were cleaned ultrasonically (Vitasonic, VITA Zahnfabrik, Bad Säckingen, Germany) in distilled water for 10 minutes. Specimens were then embedded in plastic moulds (diameter: 12 mm, height: 10 mm) using auto-polymerized polymethylmethacrylate (Scandiquick, Scandia, Hagen, Germany), keeping the upper surface free for bonding purposes using a device that maintained the specimens parallel to the x-axis. The specimens were then randomly divided into 3 main groups to be contaminated and conditioned:

Surface contamination and conditioning methods

Group C: This group received no contamination on the specimen surfaces and acted as the control group.

Group S: Artifical saliva (Biofórmula, São José dos Campos, São Paulo, Brazil) (pH=6) was rubbed on the specimen surfaces one coat using a microbrush and rinsed with water spray for 15 s and air-dried for 10 s according to a previously described protocol [29,30].

Group S+AA: Air-abrasion was performed using aluminum oxide particles coated with silica (30 µm, CoJet Sand, 3M ESPE) at 2.8 bar pressure from a distance of approximately 10 mm from the surface, in a circular motion for 15 s using an intraoral air-abrasion device (Dento-Prep, RØNVIG, Daugaard, Denmark) and subsequently, specimen surfaces were saliva contaminated as described in Group S.

Half of the specimens in each group were not etched and the other half were etched with 37.5% H_3PO_4 (Ultraetch, Ultradent, Utah, USA) for 60 s.

Adhesion procedures

The zirconia specimen surfaces in each group were silanized (Monobond Plus, Ivoclar, Vivadent, Schaan, Ivoclar) for 60 s, air-dried and were further divided into two groups depending on the luting cements based on methacrylate (VL, Variolink II, Ivoclar Vivadent) and 10-MDP monomer (PN, Panavia 21, Kuraray) to be bonded onto the specimens.

One calibrated operator carried out adhesive procedures throughout the experiments. Translucent polyethylene molds (height: 4 mm, diameter: 1 mm) were stabilized on the zirconia specimens in a custom made device. Base and catalyst paste of dual polymerized resin cements were mixed in a 1:1 ratio on a mixing pad for 10 s. The mold was filled with the resin cement; a metal pin was inserted into the mould, pushing the cement to the substrate surface and ensuring a thickness of 100 µm at the first layer of cement, simulating an acceptable clinical cement film thickness [31]. VL cement was photo-polymerized using an LED unit (Bluephase G2, Ivoclar Vivadent) for 40 s from 5 directions from a distance of 2 mm. Light intensity was assured to be higher than 1200mW/cm^2, verified by a radiometer after every 10 specimen (Model 100, Kerr, Orange, CA, USA). Oxygen inhibiting gel (Oxyguard, Kuraray) was applied at the bonded margins and rinsed with cupious water after 1 minute.

Polyethylene molds were gently removed from the test specimens. Half of the specimens were kept dry at 37ºC for 24 h in dark and the other half was subjected to thermocycling for 5000 cycles between 5 and 55°C in distilled water (Haake DC 10, Thermo Haake, Karlsruhe, Germany). The dwelling time at each temperature was 30 s and the transfer time from one bath to the other was 10 s.

Microshear tests

For the microshear bond test (MSB), specimens were mounted in the jig of the Universal Testing Machine (Zwick ROELL Z2.5 MA 18-1-3/7, Ulm, Germany) and the shear force was applied using a shearing blade to the adhesive interface until failure occurred. The load was applied to the adhesive interface as close as possible to the surface of the substrate at a crosshead speed of 0,5 mm/min and the stress-strain curve was analyzed with the software program (TestXpert, Zwick ROELL, Ulm, Germany).

Microscopic examination and failure analysis

After adhesion tests, debonded specimen surfaces were examined in order to analyze the failure types using an optical microscope (Zeiss MC 80 DX, Jena, Germany) at x50 magnification. Failure types were planned to be classified as follows: Score 1: Adhesive failure at ceramic-cement interface with no cement remnants left on the substrate, Score 2: <1/3 cement left adhered on the substrate, Score 3: >1/3 cement left adhered on the substrate, Score 4: Cohesive failure within the substrate.

Statistical analysis

A sample size of 10 in each group was calculated to have more than 80% power to detect a difference in means of 8 MPa between groups with a standard deviation of 5 MPa using a two-group Satterthwaite t-test (SPSS Software V.13 for Windows, Chicago, IL, USA) with a 0.05 two-sided significance level. Statistical analysis was performed using Statistica 8.0 software for Windows (StatSoft, Inc., Tulsa, OK, USA). Power analysis and Kolmogorov-Smirnov and Shapiro-Wilk tests were used to test normal distribution of the data. As the data were normally distributed, Univariate analysis of variance was applied to analyze possible differences between the groups where the bond strength was the dependent variable and contamination and conditioning (3 levels: C; S+R; S+AA), etching (without and with 37% H_3PO_4), cement types (2 levels: methacrylate-based; MDP-based) and aging types (2 levels: dry versus thermocycle) as independent variables. Due to significant differences between groups, multiple comparisons were analyzed using Tukey's, Bonneferroni and 2-sided Dunnett-T post hoc tests. Maximum likelihood estimation without a correction factor was used for 2-parameter Weibull distribution to interpret predictability and reliability of adhesion (Minitab Software V.16, State College, PA, USA). P values less than 0.05 were considered to be statistically significant in all tests.

Results

Debonded specimens during thermocycling were considered 0 MPa.

While saliva contamination, clraning method ($p<0.001$) and aging ($p<0.05$) significantly affected the bond results, cement type did not show significant difference after aging ($p>0.05$).

Bond strength of PN (1.2-4.4 MPa) on saliva contaminated and etched zirconia was more stable than that of VL (0-2.8 MPa) (Table 2).

After saliva contamination and etching, in the aged groups PN presented higher moduli in all groups (C: 4.5; S: 2.5; S+AA: 6.6) compared to that of VL (C: 0; S: 0; S+AA: 2.9) (Table 2).

After aging, bond strength results decreased the most with VL (3 to 100%) compared to PN (32 to 71%) but the decrease was less in the air-abraded groups after aging (VL: 3%; PN: 32%) (Figs. 2a-d).

Exclusively adhesive failures were experienced in all groups.

Discussion

This study evaluated the effect of cleaning regimens to remove saliva contamination from zirconia surface in order not to impair the adhesion of the resin cement after aging. Based on the results of this study, since cement chemistry, cleaning method affected the results, the null hypothesis could be rejected.

Previous studies using stress distribution analyses have reported that some of the bond strength tests do not appropriately stress the interfacial zone [32,33]. Shear tests have been criticized for the development of non-homogeneous stress distributions at the bonded interface, inducing either underestimation or misinterpretation of the results, as the failure often starts in one of the substrates and not solely at the adhesive zone [32,33]. Conventional tensile tests also present some limitations, such as the difficulty of specimen alignment and the tendency for heterogeneous stress distribution at the adhesive interface. On the other hand, when specimens are aligned correctly, the microtensile test shows more homogeneous distribution of stress, and thereby more sensitive comparison or evaluation of bond performances [32]. However, minute deviations in specimen alignment in the jig may cause increase bond strength due to shear component being introduced during debonding the adhered joints [32]. In this study, microshear test was used that eliminates the problems of pre-test failures prior to tesing as in the case of microtensole test and avoids problems related to shrinkage in macroshear tests due to large bonded surface area.

The results obtained correspond to the ranges summarized in a recent meta-analysis with similar cements [10]. Especially with the MDP based cement (PN), higher results were reported using the macrotensile test even in prolonged aged conditions [8]. However, it has to be noted that in those studies, cements were additionally polymerized in an oven under heat that was not practiced in this study as heat polymerization is neither manufacturer`s recommendation nor clinically relevant. Nevertheless, the results achieved on zirconia even in the control group of this study are still much inferior than those reported for glassy matrix ceramics after etching with hydrofluoric acid and silanization [4,5].

In this study, one methacrylate, one MDP-based cement was used. Such cements contain multifunctional phosphoric acid dimethacrylate modified monomers in their chemical compositions [10]. As zirconia ceramic includes oxides, in principle, the surface conditioning with silane coupling agents like the one used in this study having adhesive functional monomers such as phosphoric acid group monomer in their composition are expected to improve the bonding to zirconia. However, after aging conditions, drastic decrease was observed in bond strength of both cements being more significant for mechnacrylate-based cement. Likewise, based on the exclusive incidences of adhesive failures with both cements it cannot be stated that sufficient bond could be established to zirconia.

Oral fluids are known to degragade ceramic-resin interfaces resulting in slow crack growth [34]. Testing the adhesive joints either after water storage or thermocycling yield to hydrolytic degradation at the interface and usually results in decreased bond strength of resin-based materials to zirconia [7,34].

The mechanism of phosphoric acid is not completely understood but it is postulated that the acid possibly penetrates the salivary film and etches the porcelain surface underneath, thereby releasing the salivary film from the surface [27]. Phosphoric acid also removes the adsorbed proteins by coagulation or desorption from ceramic surface onto the cleaning particles [30]. Subsequent water rinsing can then remove the coagulated or desorbed proteins. However, phosphoric acid passivates the zirconia surface when used in combination with phosphate methacrylate based primers used in adhesive cementation and decrease bond strength [35,36]. In this study, methacrylate based resin cement was also used. Apparently, phosphoric acid did not hinder the copolymerization between silane and the methacrylate resin cement tested when bond strength results in dry conditions are considered. However, after aging conditions drastic decrease and practically no bond strength was achieved with the methacrylate-based resin cement. In return, when Weilbul parameters are considered, 10-MDP based cement showed comparably more reliable results. These parameters could ve verified on a larger sample but due low bond strength data, this may also now add too much since low adhesion to zirconia is well-known [10]. Depending on the test method, the numeric values of the bond strength with this type of cement varies but there is general consensus that chemically polymerized 10-MDP resin cement provides more durable adhesion to zirconia which was also verified with the microshear test method in this study. Saliva proteins bound to the ceramic surface could not be completely removed from the specimens. In previous studies, the use of the cleaning paste including zirconia particles was proved to be an effective method to restore bond capacity of the saliva contaminated glass-based ceramic [37,38]. Such pastes has been tried on zirconia in several studies to remove saliva contamination [39-44,] with the outcome that cleaning could restore the lost adhesion but this is also in part cement product dependent [43]. This aspect requires further investigations as cleaning may be of less importance for some cement types. Yet, all these studies were in agreement that saliva contamination impairs adhesion to zirconia and the obtained bond strength results were not above 10 MPa.

Nevertheless, in case of saliva contamination after the air-abrasion is achieved at the laboratory, the bond strength was less impaired. This could be attributed to the surface roughness created after air-abrasion that possibly compensates partially for the passivating effect of the saliva and subsequent etching. However, even in silica-coating was practiced, it has been previously reported that the bond achieved is also prone to hydrolytic degradation [10,45].

The possible saliva contamination during the intraoral try-in procedures could impair bond strength results and indicates the clinical significance of this investigation particularly for minimal invasive reconstructions. Adhesive cementation protocols should consider removal of contamination media that is a critical step to avoid debonding. In addition, etching of the surface for removal of saliva further decreases the adhesion of resin cements and this should be avoided in adhesion protocols.

Conclusions

From this study, the following could be concluded:

(1) Saliva contamination and aging decreased the adhesion of both methbacrylate and 10-MDP based resin cements to zirconia.

(2) After aging, bond strength results decreased the most with methacrylate-based cement compared to 10-MDP based resin cement.

(3) Etching non-contaminared or saliva contaminated zirconia surfaces with 37.5% H_3PO_4 practically resulted in no adhesion for methacrylate-based cement but the effect was less in the air-abraded groups.

(4) Regardless of the contamination and cleaning methids emoployed, exclusively adhesive failures were experienced in all groups.

Clinical Relevance

Saliva contamination decreases adhesion of resin cements to zirconia. Cleaning the contaminated surfaces with 37.5% H_3PO_4 inhibits adhesion and therefore should be avoided in adhesive cementation of zirconia reconstructions. Air-abrasion of zirconia surfaces and the use of 10-MDP based chemically polymerized resin cement delivers the best adhesion.

Acknowledgement

We would like to acknowledge the manufacturing companies for generous provision of resin cements used in this study and Mr. A. Trottmann for his assistance during the experimental procedures.

Conflict of interest

The authors did not have any commercial interest in any of the materials used in this study.

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Captions to figures and tables:

Tables:

Table 1. The brands, abbreviations, manufacturers, chemical compositions and batch numbers of the materials used in this study.

Table 2. The mean microshear bond strength values (MPa ± standard deviations), Weibull parameters (shape and scale), distribution and frequency of failure types per experimental group analyzed after bond strength test: Score 0: Adhesive failure at ceramic-cement interface with no cement remnants left on the substrate, Score 1: <1/3 cement left adhered on the substrate, Score 2: >1/3 cement left adhered on the substrate. C: Control; SA: Saliva contaminated; S+AA: Saliva contaminated and air-abraded. VL: Variolink II; PN: Panavia 21.

Figures:

Fig. 1 Allocation of experimental groups based on the surface contamination and etching, resin cements and aging.

Figs. 2a-d Bond strength change in percentage after etching with 37% H_3PO_4 in groups **a)** VL-Dry, **b)** PN-Dry, **c)** VL-TC, **d)** PN-TC. *For group descriptions see Fig. 1 and Table 1.

Tables:

Brand	Manufacturer	Chemical Composition	Batch number
Variolink II (VL)	Ivoclar Vivadent, Schaan, Liechtenstein	bis-GMA, UDMA, TEGDMA, BPO, camphorquionine, barium glass, ytterbium trifluoride, Ba-Al fluorosilicate glass, spheroid mixed oxide Particle size: 0.04 - 3 μm (mean: 0.7 μm), Filler load (base: 73.4 wt%) Filler load (catalyst high viscosity: 77.2 wt%)	J 17818
Panavia 21 (PN)	Kuraray, Tokyo, Japan	Paste A: 10- Methacryloyloxydecyl dihydrogen phosphate Paste B: Hydrophobic aromatic dimethacrylate, Hydrophilic aliphatic methacrylate, Hydrophilic aliphatic dimethacrylate silanated barium glass filler (wt%)	00443 A 00221 B
CoJet	3M ESPE, St. Paul, Minnesota, USA	30 μm aluminum oxide particles coated with silica	105
Etching gel	Ultradent, Utah, USA	37.5% H_3PO_4	8561

Table 1. The brands, abbreviations, manufacturers, chemical compositions and batch numbers of the materials used in this study.

						Weibull parameters		Failure type distribution (%)
Group number	**Conditioning/ Contamination**	**Cement**	**Aging**	**Etching with 37% H_3PO_4**	**Microshear bond strength (MPa) (Mean ± SD) *(CI 95%)***	**Shape**	**Scale**	**Score 0**
1	C	PN	Dry	No	8.2 ± 1.9 *(6.8-9.5)*	4.5	8.9	100
2	C	PN	Dry	Yes	5.7± 1.4 *(4.7-6.7)*	4.6	6.2	100
3	C	PN	Aging	No	2.2 ± 2.2 *(0.7-3.8)*	1.7	3.6	100
4	C	PN	Aging	Yes	1.3 ± 2.2 *(-0.2-2.9)*	4.5	4.9	100
5	C	VL	Dry	No	6.9 ± 1.3 *(6.0-7.9)*	5.4	7.5	100
6	C	VL	Dry	Yes	5.7± 2.9 *(3.6-7.8)*	2.1	6.5	100
7	C	VL	Aging	No	2.3 ± 2.4 *(0.6-4.0)*	7.3	4.9	100
8	C	VL	Aging	Yes	0.0± 0.0 *(0-0)*	-	-	100
9	S	PN	Dry	No	6.6± 2.0 *(5.2-8.1)*	3.3	7.3	100
10	S	PN	Dry	Yes	5.3± 1.3 *(4.4-6.2)*	4.6	5.8	100
11	S	PN	Aging	No	4.2± 2.0 *(2.8-5.7)*	3.6	5.2	100
12	S	PN	Aging	Yes	1.2± 1.4 *(0.2-2.3)*	2.5	2.8	100
13	S	VL	Dry	No	4.2± 1.9 *(2.9-5.5)*	4.3	5.1	100
14	S	VL	Dry	Yes	4.7± 0.9 *(4.0-5.4)*	5.4	5.1	100
15	S	VL	Aging	No	2.3± 2.0 *(0.9-3.7)*	5.7	4.2	100
16	S	VL	Aging	Yes	0.0± 0.0 *(0-0)*	-	-	100
17	S+AA	PN	Dry	No	6.9± 1.2 *(6-0-7.8)*	5.7	7.4	100
18	S+AA	PN	Dry	Yes	6.3± 1.1 *(5.5-7.1)*	5.8	6.7	100
19	S+AA	PN	Aging	No	6.5± 1.6 *(5.4-7.7)*	4.7	7.1	100
20	S+AA	PN	Aging	Yes	4.4± 0.8 *(3.9-5.0)*	6.6	4.8	100
21	S+AA	VL	Dry	No	6.0± 0.7 *(5.5-6.4)*	12.3	6.2	100
22	S+AA	VL	Dry	Yes	4.9± 0.7 *(4.4-5.4)*	8.8	5.1	100
23	S+AA	VL	Aging	No	2.9± 2.0 *(1.4-4.3)*	2.5	4.0	100
24	S+AA	VL	Aging	Yes	2.8± 2.3 *(1.2-4.4)*	2.9	4.5	100

Table 2. The mean microshear bond strength values (MPa ± standard deviations), Weibull parameters (shape and scale), distribution and frequency of failure types per experimental group analyzed after bond strength test: Score 0: Adhesive failure at ceramic-cement interface with no cement remnants left on the substrate, Score 1: <1/3 cement left adhered on the substrate, Score 2: >1/3 cement left adhered on the substrate. C: Control; SA: Saliva contaminated; S+AA: Saliva contaminated and air-abraded. VL: Variolink II; PN: Panavia 21.

Figures:

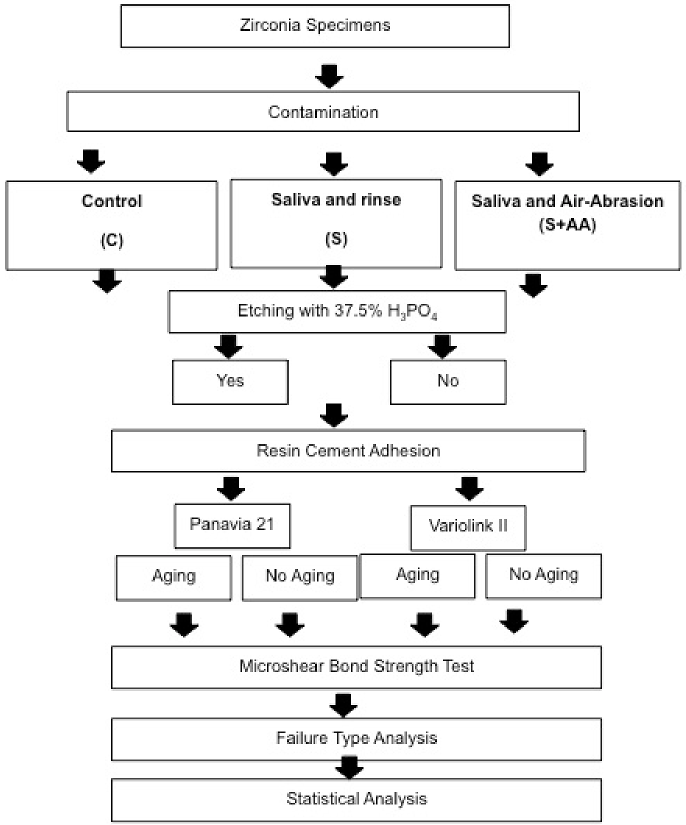
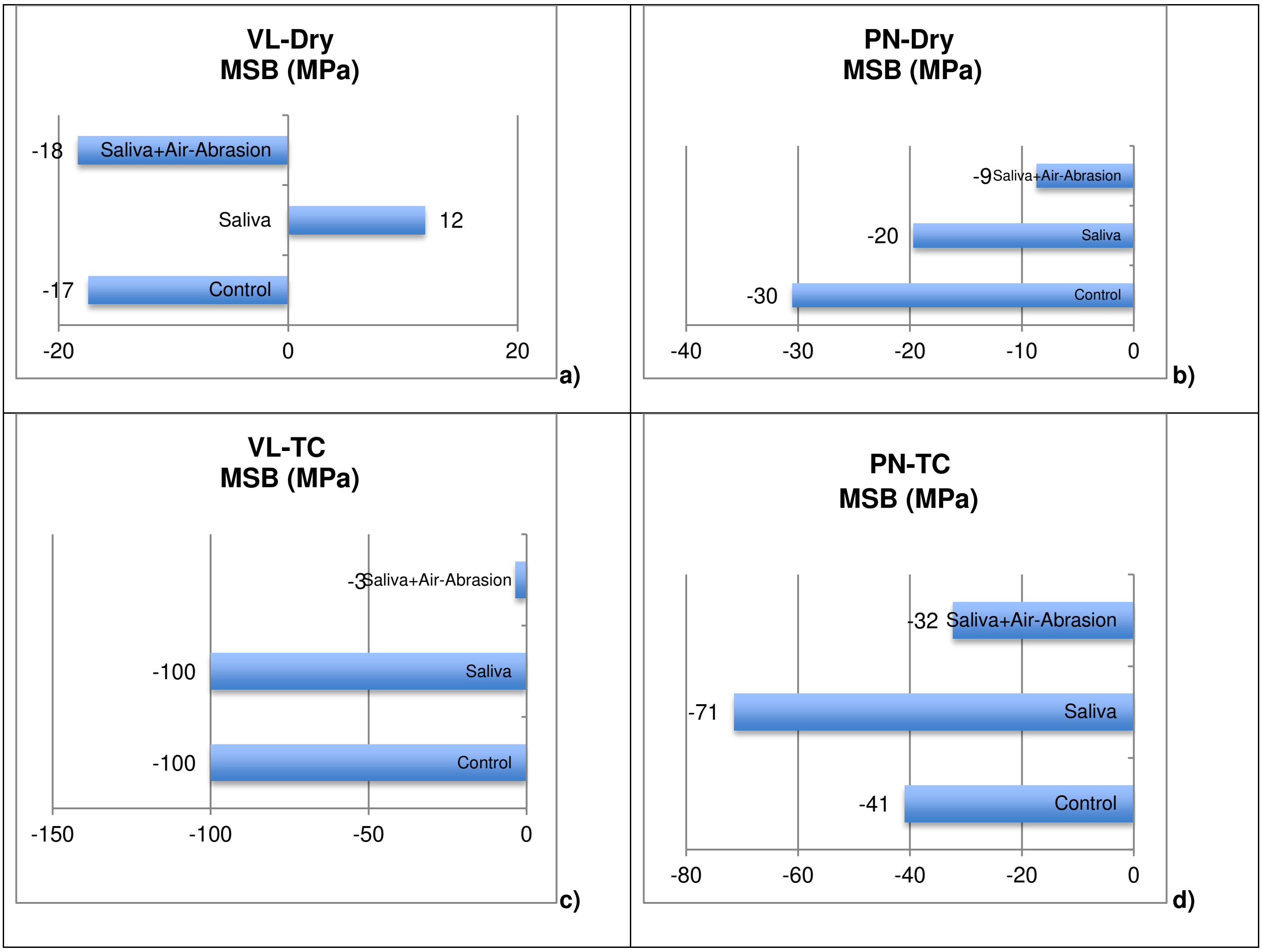


Fig. 1 Allocation of experimental groups based on the surface contamination and etching, resin cements and aging.



Figs. 2a-d Bond strength change in percentage after etching with 37% H_3PO_4 in groups **a)** VL-Dry, **b)** PN-Dry, **c)** VL-TC, **d)** PN-TC. *For group descriptions see Fig. 1 and Table 1.